MENTATION PAGE

Form Approved
OMB No. 0704-0188

REPORT DATE: October 1991

3. REPORT TYPE AND DATES COVERED: Final Report 9/1/86 - 9/30/91

A Fundamental Understanding of the Interfacila Compatibility in Hybrid Materials Systems

5. FUNDING NUMBERS: 61102F 2306/A1

2

Rene B. Testa, and John K. Tien

Columbia University
Center fcr Strategic Materials
New York, NY 10027

8. PERFORMING ORGANIZATION REPORT NUMBER: AFOSR-TR- 91 0948

AFOSR/NC
Bolling AFB, DC 20332-6448

10. SPONSORING/MONITORING AGENCY REPORT NUMBER: AFOSR-86-0312

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12b. DISTRIBUTION CODE

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15. NUMBER OF PAGES: 17

16. PRICE CODE

17. SECURITY CLASSIFICATION OF REPORT	18. SECURITY CLASSIFICATION OF THIS PAGE	19. SECURITY CLASSIFICATION OF ABSTRACT	20. LIMITATION OF ABSTRACT
UNCLASSIFIED	UNCLASSIFIED	UNCLASSIFIED	SAR

FINAL TECHNICAL REPORT

AFOSR-86-0312

UNDERSTANDING THE INTERFACIAL COMPATIBILITY OF HYBRID COMPOSITE MATERIALS

Rene B. Testa
Principal Investigator
Professor and Director
Carleton Strength of Materials Laboratory
Columbia University
New York, NY 10027

John K. Tien
Principal Investigator (Subcontract)
Rashid Engineering Regents Chair
and Director,
Strategic Materials R&D Laboratory
The University of Texas
Austin, TX 78712

October 1991

ABSTRACT

Efforts in this program have focussed on developing an understanding of the nature and kinetics of component interactions in hybrid material systems at elevated temperature. Previous work on the chemical interactions in tungsten fiber reinforced superalloys was expanded on to include the effect of matrix chemistry on fiber recrystallization kinetics. General studies to assess the kinetics and diffusion path behavior of metal/intermetallic (W/Ni_3Al) and intermetallic / intermetallic ($TiAl/Ni_3Al$) were also undertaken. A feasibility study for production of diffusion barrier layers via ion implantation were also conducted. In response to a controversy in the scientific community, the influence of fiber/matrix interactions on the anomalous creep behavior of SiC/Al was investigated.

INTRODUCTION

This program has focussed on gaining a fundimental understanding of the nature and effect of interdiffusion and reactions in hybrid materials. Prior to the inception of this program, work had been done on both the very simple tungsten fiber reinforced niobium system and on the considerably more complex tungsten fiber reinforced superally (TFRS) system [1-7]. In both cases methodologies for characterizing the high temperature behavior were developed. In the case of W/Nb the very simple solid solution behavior lended itself to numerical solutions to Fick's 2nd law, thereby enabling very long term predictions of interdiffusion behavior. Unlike the W/Nb system, TFRS composites were found to exhibit the formation of a single reaction zone phase at the fiber / matrix interface. The kinetics of this phase's formation were found to be highly dependent on matrix chemistry, and through matrix modifications the kinetics were minimized. Although a good understanding of the reaction kinetics of TFRS composites was obtained, several key issues remained to be addressed regarding the long term stability of TFRS, in particular, and all metal matrix composites (MMCs) and intermetallic matrix composites (IMCs), in general.

These remaining questions to be answered formed the framework of this program. First, although reactions at the fiber / matrix interface were considered to be a key factor in TFRS composite integrity, recrystallization of the tungsten fibers caused by nickel influx is at least as important. As a result, one of the first areas that was examined in this program was the effect of matrix chemistry on the recrystallization behavior of TFRS composites. Secondly, since the number of constitutient species in superalloys is so large, a pseudobinary approach was necessary to characterize the interdiffusion and reaction behavior. Thus, although this method was effective in characterizing the TFRS system, a more general understanding was called for. This lead to this program's work on the relatively simple ternary W/Ni_3Al metal/intermetallic system and $Ni_3Al/TiAl$ intermetallic/ intermetallic system. Thirdly, the possibiity of reducing the kinetics of interdiffusion through interface modification had not been attempted. Thus, an assessment of the feasibility of ion implanted diffusion barriers was carried out. Additionally, in response to a controversy that developed in the scientific community during the program period, a preliminary study of the relationship between the fiber/matrix interface and the anomolous creep behavior exhibited in the SiC/Al system was performed in order to confirm or deny that said anomaly was due to diffusion. Results of each of these efforts are breifly discussed below.

This program has resulted in one Ph.D. dissertation, three M.S. theses and two B.S. theses. Six technical papers were published and two are presently in preparation. Additionally, seven lectures were given at technical conferences. Copies of the published papers are attached as Appendix I.

PROGRAM RESULTS

Recrystallization Mechanisms of W-fibers in TFRS Composites

Tungsten fiber reinforced superalloys (TFRS) have long been candidates for high temperature composites allowing for significant increases in operating temperatures through increased creep resistance and strength. TFRS composites present several concerns including diffusion induced recrystallization of the tungsten fibers and reaction zone formation at the fiber/matrix interface. Recrystallization has a pronounced effect on the strength, creep resistance, and toughness of the tungsten fibers. Fiber/matrix diffusional reactions in TFRS have been shown to produce brittle intermetallic phases that, as they continue to grow, may adversely affect mechanical strength, due to loss of fiber cross-section, and toughness, from the defect sensitive intermetallic itself.

It has been previously reported that reaction zone growth is rate controlled by interdiffusion across the reaction zone phase. The interdiffusion coefficient for these systems using a pseudobinary approximation can be expressed as

$$D(T) = A\, K_{rz}(T)^{1/2}\, K_f(T)^{1/2} \qquad (1)$$

where A is a constant of proportionality dependent on the interfacial chemistries at the fiber/reaction zone and reaction zone/matrix interfaces, $K_{rz}(T)$ and $K_f(T)$ are the parabolic rate constants for the growth of the reaction zone and the growth of the portion of the reaction zone that displaces the fiber. Several matrix alloys have been ranked according to the product of the roots of these rate constants. The chemistries of these alloys and the kinetic results of this ranking are shown in Tables I and II. Alloys 89 and 90 are experimental alloys developed in an effort to minimize reaction zone growth kinetics. As can be seen, reductions of iron and cobalt decrease interdiffusion across the reaction zone and thereby decrease the kinetics of reaction zone formation. Recrystallization of the tungsten fibers reinforcement is also a primary concern for TFRS composites. Recrystallization of ThO_2 doped tungsten wires normally occurs at about 2000° C.

A number of studies have shown that a number of elements, most notably nickel, cause this recrystallization temperature to drop dramatically when they are in contact with the fibers. In fact conventional wisdom to date has been that any increases in nickel content of the matrix alloy in TFRS composites would be accompanied by increased recrystallization kinetics of the fibers in that composite system. Since very pure tungsten wires recrystallize at about the same temperature as the poisoned ThO_2 doped wires, it appears that the infusion of the poisoning elements affects recrystallization inhibiting nature of the dopant. A complication has been some uncertainty regarding the nature and distribution of dopant particles. Perhaps the most notable theory to explain the diffusion promoted recrystallization of doped tungsten wires is that the poisoning species, which is primarily diffusion along grain boundaries through short circuit paths, lowers the interfacial energy of the grain and subgrain boundaries, thereby overcoming the effect of the pinning dispersoids. However, the effect of the poisoning species on the dispersoid/bulk tungsten energy may also be playing a significant role.

Table I. Nominal chemistries in atomic percents of various TFRS matrix alloys.

Alloy	Ni	Co	Fe	Nb	Cr	Al	Ti	Mo	W
FeCrAlY	0	0	71	0	25	4	0	0	0
SS316	12	0	70	0	18	0	0	0	0
Incoloy 907	25	13	57	3	0	.1	.3	0	0
Incoloy 903	38	15	42	2	0	1	2	0	0
Waspaloy	56	13	0	0	21	3	4	3	0
Alloy 89	66	0	0	0	21	6	4	0	3
Alloy 90	64	0	0	0	21	6	4	0	5

Table II. Reaction zone growth kinetics for selected TFRS composites annealed at 1093° C.

Matrix	Fe	Fe+Co	Ni	$K_{rz}^{1/2}$ $K_f^{1/2}$ ($\times 10^{-12}$ cm^2/sec)
FeCrAlY	71	71	0	3.5
SS316	70	70	12	2.9
Incoloy 907	57	70	25	1.7
Incoloy 903*	42	57	38	0.8
Waspaloy	0	13	56	0.3
Alloy 89	0	0	66	0.05

As stated above, the conventional wisdom, with regard to matrix nickel content, has been that increases in nickel content should promote accelerated tungsten fiber recrystallization. The purpose of this investigation pivoting about alloys #89 and #90 is to attempt to illucidate the previously reported matrix chemistry effect.

Composite samples were fabricated by powder metallurgical techniques employing 1.5% ThO_2 doped tungsten wires that were nominally 4 mils (100 micrometers) in diameter. The fibers were unidirectionally oriented and constituted approximately 40 volume percent of the composite. Composite samples were annealed in vacuum at temperatures from 1050° C to 1200° C for 10 to 500 hours. Samples were metallographically prepared and etched to illucidate the tungsten microstructure. Measurements of the penetration of the recrystallization front were done by optical and scanning electron microscopy.

The annealing schedule of the earlier reaction zone growth study and the limited availability of source material limited the amount of useful recrystallization data that could be obtained from the samples. In many cases, although annealing times were ideal for reaction kinetics measurements, major progressions in recrystallization were oversteped. However, some very interesting results were obtained. The FeCrAlY matrix composites did not exhibit significant recrystallization at the temperatures and exposure times studied. Figure 1 illustrates recrystallization penetration data for several other matrix materials annealed at 1100° C and 1130° C, including some of the 1100° C data of L.O.K.

Larsson. The earlier composites of Larsson show more accelerated recrystallization than the higher and lower nickel containing alloys of the present study. Further, the very high nickel alloys #89 and #90 were annealed 30° C higher than the others. The discrepancies in the data may well be explainable considering the assumed differences in processing, and therefore thermal, histories and experimental error. Additionally, although considerably more nickelis available in the matrix of these alloys to source diffusion induced recrystallization, these alloys were specifically designed to minimize diffusion across the anticipated reaction zone. Thus, there may be some level of competition between total nickel availability and interdiffusional kinetics that provide nickel to the fiber surface.

Based on the current study's data, it appears that the "conventional wisdom" regarding the effect of increased nickel content in TFRS composite matrices on recrystallization behavior may not be as important as previously thought. Although the complete absence of nickel does dramatically decrease recrystallization kinetics (as demonstrated by the FeCrAlY matrix system), the current study indicates that once a threshold level of nickel is reached, little effect, if any, may be seen on the recrystallization behavior. It is proposed that the cause of any observed effect may be accredited to the total amount of nickel available in the matrix and the transport kinetics of nickel across the reaction layer. For this reason, among others, each and every

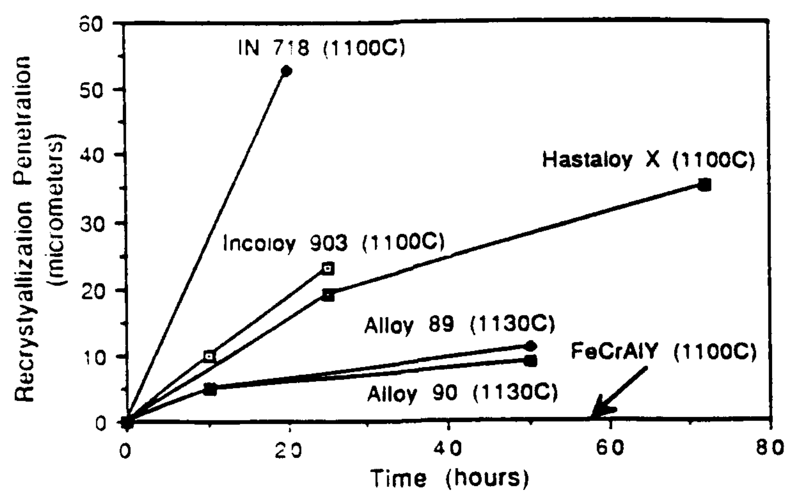


Figure 1. Recrystallization front penetration vs. time for various TFRS composites annealed at 1100° C and 1130° C.

candidate TFRS system must be evaluated individually since a priori estimations of the relative effects of each of these competing factors appears to be difficult.

Interdiffusion in the W/Ni_3Al System

In this aspect of the program, the interdiffusional behavior of the W / Ni_3Al system was evaluated. This system was chosen in order to study interdiffusion in a relatively simple intermetallic matrix composite without the complications of ternary intermetallic intermediate phases. Diffusion couples were fabricated by hot pressing commercially available tungsten sheet and a polycrystalline IC-72 Ni_3Al alloy. Hot pressing was performed at 1000C for 2 hours at approximately 9.4 ksi. Although this particular Ni_3Al alloy incorporates small amounts of B and Hf, they are not believed to be in sufficient enough quantity to significantly affect the bulk interdiffusion behavior.

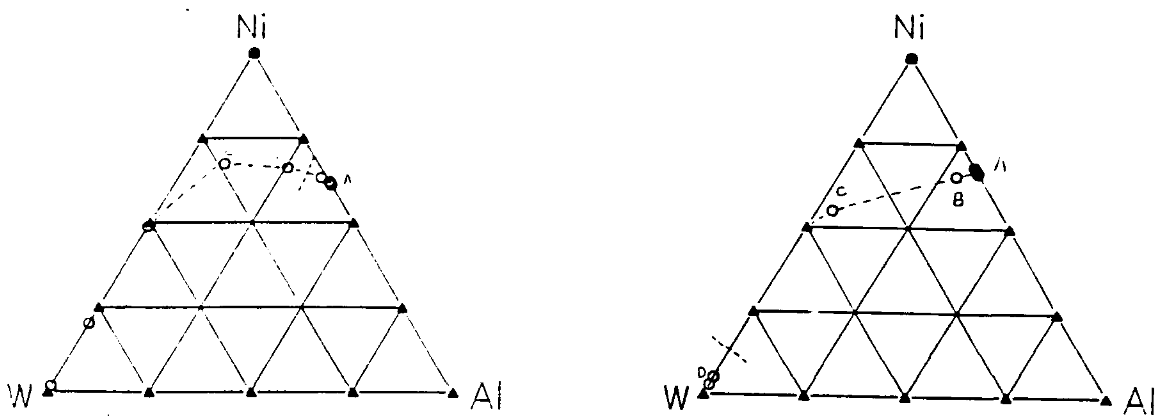
The couples were subsequently annealed in vacuum in accordance with the schedule shown in Table III. Composition-position profiles and diffusion path maps were determined for the various diffusion couples using SEM/EDS at 1 micron intervals across the interdiffusion zone.

TABLE III

Annealing Schedule for the W / Ni_3Al Specimens

Temperature (K)	Time (Hrs)
1300	10
1300	25
1300	100
1300	400
1400	10
1400	25
1400	100
1400	400

Diffusion path maps for 1300K and 1400K are shown in Figure 2a and 2b. The 1400K calculated isotherm of Kaufman and Nesor is shown in Figure 3. Note that Ni and W appear to be the prime diffusing species with virtually no Al flux. None of the diffusion couples examined evidenced the growth of a classical interphase at the fiber/matrix interface. Rather, the flux of Ni and W seems to have promoted fine precipitation of Ni_4W in both of the terminal phases adjacent to the interface. Although a full kinetic analysis has not yet been completed, phase evolution in this system appears to be controlled by the kinetics of formation of the Ni_4W phase.



(a) (b)

Figure 2. Diffusion path maps for W/Ni_3Al annealed at (a) 1300K for 400 hours and (b) 1400K for 10 hours

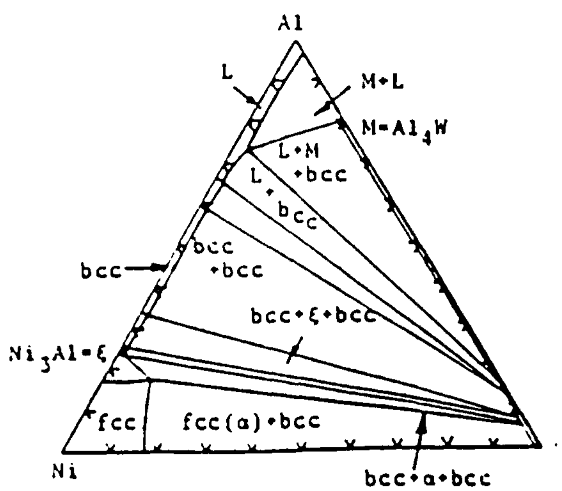


Figure 3. Calculated Ni-Al-W 1400K isotherm
[L. Kaufman and H. Nesor, <u>Can. Met. Q.</u> <u>14</u> (3)1975.]

Interdiffusion in the Ni_3Al / TiAl System

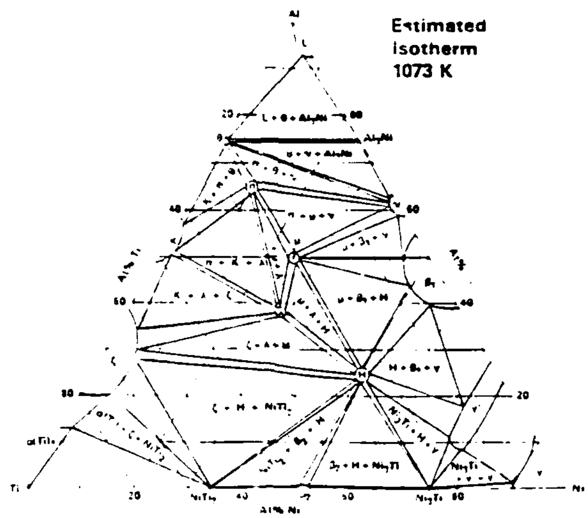
Although efforts on this aspect of the program were expected to be very extensive, difficulty in obtaining material and in having diffusion couples fabricated greatly limited the scope of work. Although there was considerable uncertainty as to the path of interdiffusion in this system, some clues could be gleaned from the ternary phase diagram. Ternary isotherms for 800C and 1027C are shown in Figure 4. Clearly, it is expected that H-phase would certainly form, but differing diffusion paths might lead to different phases being present (e.g. β_1 or ξ).

To date diffusion couples have been hot pressed of polycrystalline IC-72 Ni_3Al(B,Hf) and arc-cast TiAl. Significant reaction between the Ni_3Al and TiAl was seen in the as-received material. A SEM micrograph of the interface region is shown in Figure 5. Diffusion profiles were taken in the same manner as for the W/Ni_3Al system. The profile for the as-received material is shown in Figure 6. The interphases that were formed have been identified as follows in order from the TiAl phase to the Ni_3Al phase: κ (TiAl), ξ (Ti_3Al), λ (NiAlTi), H (Ni_2AlTi), β_1 (NiAl), γ' (Ni_3Al). The alternating noise in the profile to the immediate left of the Ni_3Al terminal phase are lamellar Al-rich and Al-poor Ni_3Al phase areas.

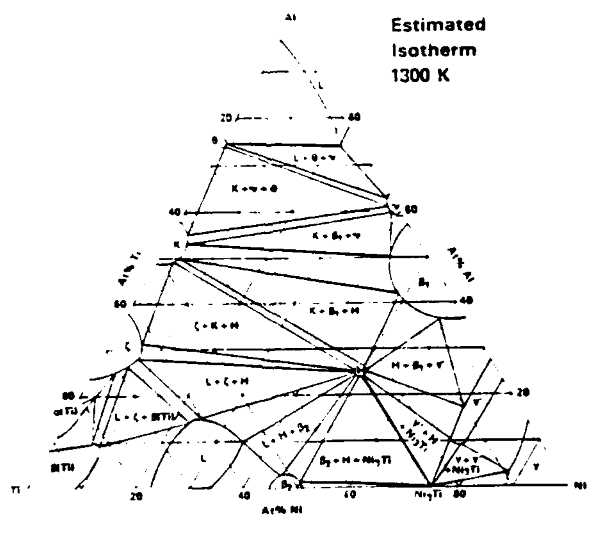
Work is continuing on this aspect of the program with other financial support for the completion of Mr. Kopp's doctoral dissertation. This work should be completed later this year.

Ion Implanted Diffusion Barriers

In an effort to investigate means by which interdiffusion between fiber and matrix components can be halted, or at least slowed, work has been performed to evaluate the feasibility of ion implanted diffusion barriers. The Pauling rules dictate that materials with considerable differences in atomic radii, valence, or electronegativity will have little solubility in one another, or will have insufficient driving force for significant interdiffusion. Based on this principle it might be expected that interdiffusion in composites may be impeded by creating a layer at the fiber/matrix interface that is very different from either of the two components.



(a)



(b)

Figure 4. Ternary isotherms for the Ni-Al-Ti system for (a) 800C and (b) 1027C.

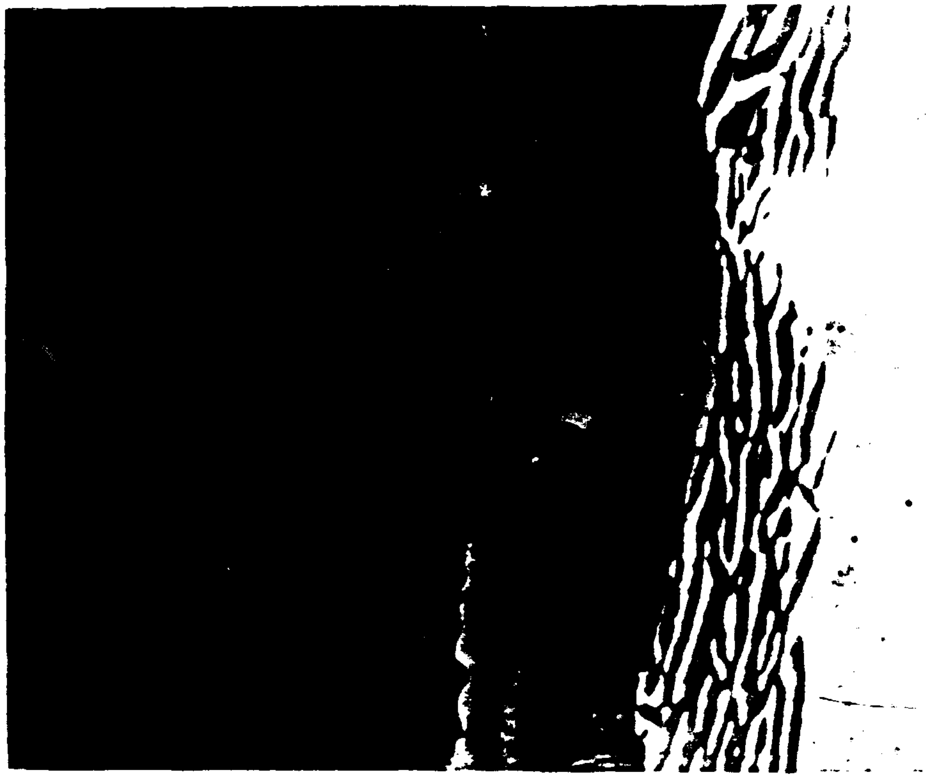


Figure 5. As received micrograph of the TiAl / Ni_3Al interface region

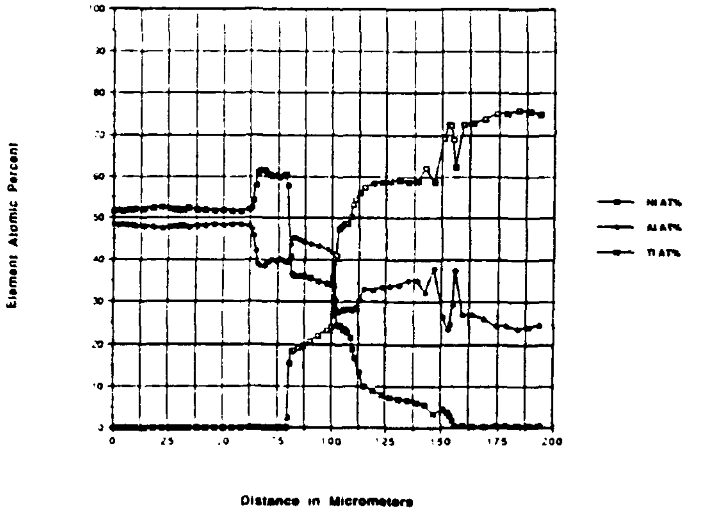


Figure 6. Composition - position profile for the as-received TiAl / Ni_3Al system

This work focussed on using ion implantation to affect such a change at the fiber/matrix interface

The simple binary W/Ni system was chosen for this study. Diffusion couples were fabricated using tungsten implanted with four different ions and ion beam sputtered nickel. Implanted ions included Na, Ba, Ca and K. Each interface received 6.4 E-11 ions/sq. cm. implanted with an accelerating potential of 190 kV. The substrate temperature was estimated to be about 700K over the 70 hour deposition time to deposit about 100 microns of nickel. These diffusion couples, along with an unimplanted control couple were annealed at 1500K for 50 hours. Composition-position profiles were obtained by SEM/EDS at 1 micron intervals across the interface.

The profiles for Na and Ca implanted barriers are shown with the as-received and control profiles in Figures 7 and 8. The evident "saturation" of W through the Ni matrix can be attributed to the very significant grain boundary diffusion that took place during the Ni deposition. Although this problem makes quantitative analysis impossible, qualitative analysis indicates that the Ca barrier appears to have indeed slowed the interdiffusion process. Na showed little effect. Both K and Ba showed increased interdiffusion.

A possible explanation of the acceleration effect is that the distortion of the larger ions may have caused some sort of dynamic recovery effect. This might be eliminated if lower ion doses were used. Similarly, the effect of the Ca barrier might be optimized by variation of the implant dose and accelerating potential.

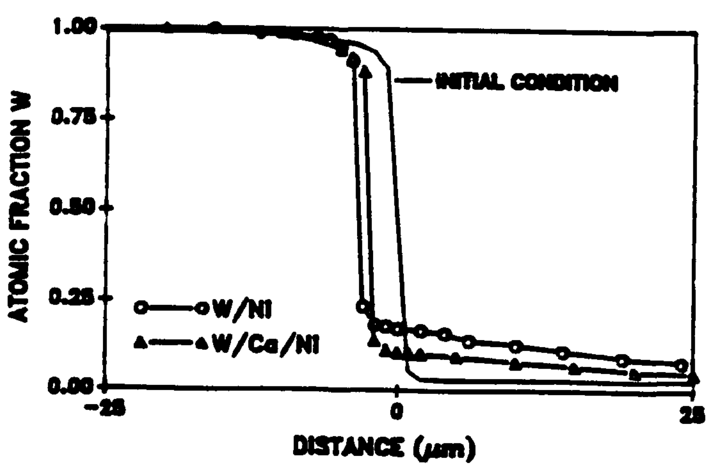


Figure 7. Composition profile for W-Ni diffusion couple with and without an ion implanted Ca Layer

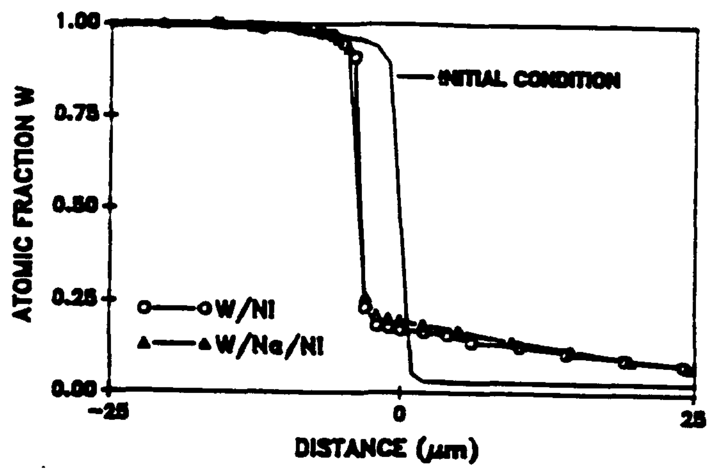


Figure 8. Composition profile for W-Ni diffusion couple with and without an ion implanted Na Layer

The Anomalous Creep of SiC/Al

Although not originally intended for this program, in response to a controversy in the community, work was performed on determing the cause of the anomolous creep behavior of a discontinuous SiC fiber reinforced Al composite. Some early work in the creep of SiC reinforced 2124 and 6061 aluminum found anomalous behavior in that no steady state region was found to exist. Other researchers had indeed found a steady state regime. This work was intended to illucidate the factors which might yield anomalous behavior and determine the influence that the fiber / matrix interface has on this behavior.

The tests performed in this study used at 20 volume percent SiC whisker reinforced 2021 alloy. Constant load creep tests were performed at temperatures below 200C up to 300C. Stresses ranged from 240 to 315 MPa at the lower temperature and from 75 to 100 MPa at the higher temperature.

Figure 9 shows plots of strain versus time on load for four stresses at 288C. Clearly, the two lower stresses exhibit classical behavior with a long steady state (minimum creep rate) regime. In contrast, the two higher stresses showed behavior where the creep rate increased monotonically until failure. Similar behavior was found for the other temperatures tested.

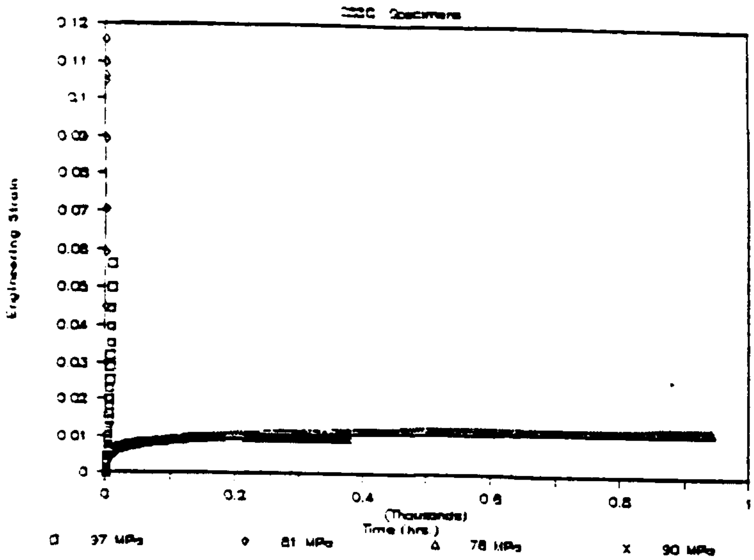


Figure 9. Strain vs. time on load for SiC / 2021 composites at 288C.

Although unconfirmed, it is hypothesized that the observed threshold stress is the stress necessary to nucleate critical sized voids at the fiber / matrix interface. Microscopic evaluations have shown a propensity for these voids to form at the corners of the SiC whiskers above the observed threshold.

CONCLUDING REMARKS

The results obtained for the W-fiber recrystallization study have shown that, in the case of TFRS, attempts to minimize one degradation mechanism is, at least partly, offset by increased kinetics of the other. However, this is not to say that some optimum matrix chemistry could not be engineered. Efforts in the metal / intermetallic and intermetallic / intermetallic systems has yielded interesting results. As stated above, this work is continuing with private funding and should be completed shortly. It is anticipated that the desired effect of being able to characterize the kinetics for these systems with closed form solutions (i.e. without having to resort to numerical methods) will be realized. The proven feasibility of ion implanted diffusion barriers is exciting. Although significant work would be required by industry to optimize implantation conditions, significant gains in limitting fiber / matrix interactions should be attainable. Finally, with respect to the anamalous creep behavior of SiC/Al, although the fiber / matrix interface is believed to play a role in the degradation mechanism, chemical interactions were ruled out as a cause.

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